



United States Environmental Protection Agency
Office of Environmental Measurement & Evaluation
11 Technology Drive
North Chelmsford, MA 01863-2431

Laboratory Report

June 29, 2011

Todd Borci - Mail Code OES04-4
US EPA New England RI

Project Number: 11060012
Project: RI DEM / EPA Sampling
Analysis: HPLC/MS/MS Source Tracking Analysis
Analyst: Peter Philbrook *PEP* 6-29-2011

Analytical Procedure:

All samples were received and logged in by the laboratory according to the USEPA New England Laboratory SOP for Sample Log-in.

Sample preparation and analysis was done following the EPA Region I SOP, EIA-LCMS_STA.0.

The SOP is based on an EPA Regional Analytical Method developed at the EPA New England Laboratory.

Date Samples Received by the Laboratory: 06/08/2011

Data were reviewed in accordance with the internal verification procedures described in the EPA New England OEME Chemistry QA Plan.

Results relate only to the items tested or to the samples as received by the Laboratory. This analytical report shall not be reproduced except in full, without written approval of the laboratory.

Report may contain multiple sections and each section will be numbered independently.

If you have any questions please call me at 617-918-8340

Sincerely,

Daniel N Boudreau 7/5/11
Daniel N Boudreau
Chemistry Team Leader

DATA QUALIFIERS

RL	Reporting limit
J	Estimated value
E	Estimated value exceeds the calibration range
L	Estimated value is below the calibration range
B	Analyte is associated with the lab blank or trip blank contamination.
R	No recovery was calculated since the analyte concentration is greater than four times the spike level.
ND	Not Detected above Reporting limit
NA	Not Applicable due to high sample dilutions or sample interferences
ME	Matrix Effect - Sample matrix was responsible for either enhanced or suppressed ionization within the electrospray ionization probe

NARRATIVE

Aqueous samples (500mL) were extracted using a solid phase extraction (SPE) technique, following EPA Method 1694, in which samples were passed through a cartridge containing a solid sorbent material which pre-concentrates the target compounds onto the sorbent. The target compounds (TCs) were then eluted off the sorbent material using methanol. The resulting eluant is concentrated to dryness and re-constituted to a final volume of 1 mL with 20/80 Methanol/Water.

A 5uL aliquot of the sample extract was injected into a High Performance Liquid Chromatograph (HPLC), and the TCs were separated chromatographically using a C8 HPLC column running a methanol / water gradient. The ionization mode used was electrospray with the polarity operating in the positive mode. The TCs were detected using a Waters Acquity TQD Tandem Quadrupole Mass Spectrometer. The tandem quadrupole is used to perform multiple reaction monitoring (MRM) where the precursor ion of interest is fragmented to product ion(s).

Quantitation was performed by the internal standard calibration method using isotopically labeled analogues. Sulfamethazine *13*C6 and Primidone *d*5 were used as a surrogate compounds to monitor extraction efficiency.

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RI DEM / EPA Sampling
HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	SD-239	Lab Sample ID:	AB18390
Date of Collection:	6/8/2011	Matrix:	Water
Date of Extraction:	06/09/2011	Final Volume:	1 mL
Date of Analysis:	06/16/2011	Extract Dilution:	1
Volume Extracted:	180 mL	pH:	N/A

CAS Number	Compound	Concentration ng/L	RL ng/L	Qualifier
611-59-6	1,7-Dimethylxanthine	27	5.6	
103-90-2	Acetaminophen	83	5.6	
29122-68-7	Atenolol	ND	5.6	
58-08-2	Caffeine	470	11.2	
298-46-4	Carbamazepine	ND	1.1	
486-56-6	Cotinine	43	1.1	
57-68-1	Sulfamethazine	ND	1.1	

Surrogate Compounds	Recoveries (%)	QC Ranges
Primidone d5	41	23 - 181
Sulfamethazine 13C6	27	15 - 132

Comments:

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RI DEM / EPA Sampling
HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	SD-239-B	Lab Sample ID:	AB18391
Date of Collection:	6/8/2011	Matrix:	Water
Date of Extraction:	06/09/2011	Final Volume:	1 mL
Date of Analysis:	06/16/2011	Extract Dilution:	1
Volume Extracted:	175 mL	pH:	N/A

CAS Number	Compound	Concentration ng/L	RL ng/L	Qualifier
611-59-6	1,7-Dimethylxanthine	42	5.8	
103-90-2	Acetaminophen	84	5.8	
29122-68-7	Atenolol	ND	5.8	
58-08-2	Caffeine	530	11.6	
298-46-4	Carbamazepine	ND	1.2	
486-56-6	Cotinine	41	1.2	
57-68-1	Sulfamethazine	ND	1.2	

Surrogate Compounds	Recoveries (%)	QC Ranges
Primidone d5	32	23 - 181
Sulfamethazine 13C6	9	15 - 132

Comments: The Sulfamethazine surrogate recovery is below the QC limit. Surrogate recovery for Primidone is within specification.

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RI DEM / EPA Sampling
HPLC/MS/MS Source Tracking Analysis

Client Sample ID:	Evergreen	Lab Sample ID:	AB18392
Date of Collection:	6/8/2011	Matrix:	Water
Date of Extraction:	06/09/2011	Final Volume:	1 mL
Date of Analysis:	06/16/2011	Extract Dilution:	1
Volume Extracted:	500 mL	pH:	N/A

CAS Number	Compound	Concentration ng/L	RL ng/L	Qualifier
611-59-6	1,7-Dimethylxanthine	12	2.0	
103-90-2	Acetaminophen	ND	2.0	
29122-68-7	Atenolol	ND	2.0	
58-08-2	Caffeine	22	4.0	
298-46-4	Carbamazepine	1.5	0.4	
486-56-6	Cotinine	6.2	0.4	
57-68-1	Sulfamethazine	ND	0.4	

Surrogate Compounds	Recoveries (%)	QC Ranges
Primidone d5	88	23 - 181
Sulfamethazine 13C6	31	15 - 132

Comments:

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Upper Norwalk River - Norwalk, CT

Laboratory Blank

Client Sample ID:	N/A	Lab Sample ID:	N/A
Date of Collection:	N/A	Matrix:	Water
Date of Extraction:	06/09/2011	Final Volume:	1 mL
Date of Analysis:	06/16/2011	Extract Dilution:	1
Volume Extracted:	500 mL	pH:	N/A

CAS Number	Compound	Concentration ng/L	RL ng/L	Qualifier
611-59-6	1,7-Dimethylxanthine	ND	2.0	
103-90-2	Acetaminophen	ND	2.0	
29122-68-7	Atenolol	ND	2.0	
58-08-2	Caffeine	5.4	4.0	
298-46-4	Carbamazepine	ND	0.4	
486-56-6	Cotinine	ND	0.4	
57-68-1	Sulfamethazine	ND	0.4	

Surrogate Compounds	Recoveries (%)	QC Ranges
Sulfamethazine 13C3	50	
Primidone d5	76	

Comments: Caffeine was detected in the laboratory blank just above the reporting limit. Samples will be qualified as "B" if the value of caffeine found in the sample is < 3X the concentration found in the blank.

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MATRIX SPIKE RECOVERY

Sample ID: AB18387

PARAMETER	SPIKE ADDED ng/L	SAMPLE CONCENTRATION ng/L	MS CONCENTRATION ng/L	MS % REC	QC LIMITS (% REC)
1,7-Dimethylxanthine	120	8.8	91	68.5	7 - 120
Acetaminophen	120	ND	80	66.7	1 - 120
Atenolol	120	ND	99	82.5	40 - 146
Caffeine	240	12	191	74.6	12 - 138
Carbamazepine	24	ND	22	91.7	27 - 144
Cotinine	24	2.4	21	77.5	48 - 131
Sulfamethazine	24	ND	14	58.3	30 - 130

LABORATORY DUPLICATE RESULTS

Sample ID: AB18386

PARAMETER	SAMPLE RESULT ng/L	SAMPLE DUPLICATE RESULT ng/L	PRECISION RPD %	QC LIMITS
1,7-Dimethylxanthine	11	8.7	23.4	50
Acetaminophen	1.7	1.3	26.7	50
Atenolol	ND	ND	ND	50
Caffeine	17	23	30.0	50
Carbamazepine	7	6.9	1.44	50
Cotinine	2.0	1.9	5.13	50
Sulfamethazine	ND	ND	ND	50
Urobilin		ND	ND	50

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LABORATORY FORTIFIED BLANK (LFB) RECOVERY

COMPOUND	SPIKE ADDED ng/L	LFB CONCENTRATION ng/L	LFB RECOVERY %	QC LIMITS (% REC)
1,7-Dimethylxanthine	120	128	107	14 - 155
Acetaminophen	120	115	95.8	43 - 129
Atenolol	120	125	104	45 - 136
Caffeine	240	220	91.7	57 - 132
Carbamazepine	24	22	91.7	39 - 136
Cotinine	24	21	87.5	60 - 127
Sulfamethazine	24	11	45.8	30 - 130

Comments:



REGION 1

CHAIN OF CUSTODY RECORD

[illegible]

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

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